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Dried fat emulsion product and method of producing the same.

Dried fat emulsion products having improved resistance to oxidation at elevated temperatures are produced by the two stage encapsulation of oil or fat globules with a hydrophilic film forming material. An aqueous dispersion of an edible fat or oil, a hydrophilic film forming material, a carbohydrate and water is emulsified to form an oil-in-water emulsion concentrate in which fat globules are encapsulated by the film forming material. After formation of the emulsion concentrate, a second portion of a hydrophilic film forming material is added to the emulsion concentrate in an amount substantially equivalent to the amount of film former in the aqueous dispersion, to provide an additional coating layer of film forming material encapsulating the fat globules. This second portion of film forming material may be added to the emulsion prior to or subsequent to drying of the emulsion concentrate.

EP 0 385 081 A2

DRIED FAT EMULSION PRODUCT AND METHOD OF PRODUCING THE SAME

This invention relates to an improved dried fat emulsion product and to the method of producing the improved emulsion product. More particularly, the invention relates to the production of edible dried fat emulsions having improved resistance to oxidation, particularly at elevated temperatures.

Dried fat emulsions have found wide acceptance in numerous convenience food products such as cake mixes, powdered shortenings, dried coffee whiteners, flavorings, topping mixes, sauce mixes, dried beverage mixes, and the like. Typically such dried emulsions are prepared by emulsifying fat or oil in an aqueous dispersion or solution of an edible hydrophilic film forming material, such as proteinaceous materials, hydrocolloid gums, starches, carboxymethylcellulose, and the like, to form an oil-in-water emulsion concentrate which is then dried, such as by spray drying. In the dried emulsion, fat globules are encapsulated by a layer of the film forming material. Such dried fat emulsions provide the advantages of ease of handling and incorporation with other dry ingredients in the preparation of various food products. Moreover, since fats are susceptible to oxidative deterioration in the presence of air, causing development of rancid odor and taste, encapsulation of the fat globules by the film forming material retards rancidity development of the fat and thereby provides the fat containing product with a longer shelf life.

Frequently there is a need for dried fat emulsion products having greater resistance to oxidation, and hence a longer shelf life, than can be obtained by the procedure described above. However, prior procedures intended to produce dried fat emulsions having increased resistance to oxidative deterioration have not been entirely satisfactory. For example, one approach to inhibit the development of oxidative rancidity in dried fat emulsion products has been to use highly saturated fats, such as hydrogenated lauric-containing fats, as the fat component. However, the use of such fats is objectionable due to dietary concerns regarding such highly saturated fats, and difficulty in reconstituting the dried emulsion, particularly in cold water. Another approach has been to incorporate antioxidants, such a butylated hydroxytolune in the fat to inhibit the oxidative process. While the use of such antioxidants is effective in extending shelf life, the use of antioxidants adds significantly to the cost of the product and is objectionable to a number of consumers.

The present invention provides a method by which dried fat emulsion products can be produced having increased resistance to oxidation and the development of rancidity, particularly at elevated storage temperatures, without requiring the use of highly saturated fats or antioxidants. In the present invention, an aqueous dispersion of an edible fat or oil, a hydrophilic film forming material, and a carbohydrate, preferably a disaccharide, and water is emulsified to form a liquid oil-in-water emulsion concentrate having a solids content of about 25% to 75% in which the fat globules are encapsulated within a continuous layer of the film forming material. After the emulsion has been formed, a second portion of a hydrophilic film forming material is added to the emulsion concentrate in an amount substantially equivalent to the amount of the film forming material contained in the aqueous dispersion, to provide an additional coating layer of film forming material encapsulating the fat globules. This second portion of the film forming material preferably is added to the emulsion concentrate prior to drying, but may, if desired be added to the emulsion concentrate after drying, such as during instantizing of the dried emulsion concentrate. The film forming material used in this second addition may be the same as or different from the film forming material used in forming the initial aqueous dispersion.

It has been found that this second addition of film forming material is surprisingly effective in improving the resistance of dried fat emulsion products to oxidative deterioration and the development of rancidity as compared to dried fat emulsion products produced by the single-stage addition of the film former, even though the total amount of film forming material included in the dried emulsion product is the same in both cases. That is, when only about one-half of the amount of hydrophilic film forming material usually used in preparing a dried emulsion product by prior procedures is used in preparing the liquid emulsion concentrate, and the remaining one-half of the film former is added after the emulsion has been formed, the dried emulsion product resulting from the two stage addition of the film forming material has significantly greater resistance to oxidation at elevated temperatures than the product obtained when all of the film forming material is included at the time the emulsion is formed. Of course, the amount of film forming material included in the aqueous dispersion prior to formation of the emulsion must be sufficient to provide a continuous film encapsulating the fat globules in the emulsion.

Other edible ingredients usually used in the production of dried fat emulsion products may, if desired, also be included in the liquid emulsion concentrate, such as, for example, stabilizers, stabilizing salts, coloring, flavoring, preservatives, vitamins, minerals, and the like, depending on the intended use of the dried fat emulsion. The dried fat emulsion product may be characterized as comprising a matrix of water

soluble constituents having as the dispersed phase therein discrete small particles of fat.

In accordance with the present invention, an aqueous dispersion is formed by mixing together an edible fat, a hydrophilic film forming material, and a carbohydrate, preferably a disaccharide, with sufficient water to provide the dispersion with a solids content in the range of about 25% to 75%. Generally, any of the edible fats and oils, of both animal or vegetable source, normally used in dried fat emulsion products may be used. Fats and oils which may be used include unhydrogenated, partially or fully hydrogenated vegetable fats and oils such as, for example, cottonseed oil, coconut oil, corn oil, soybean oil, peanut oil, sunflower oil, palm kernel oil, and the like, including mixtures thereof, as well as tallow, lard, butterfat, and the like, having the flavor, melting point range, saponification value and other characteristics desired in the product in which the dried fat emulsion is to be used. The fat comprises the major solids component of the aqueous dispersion and may comprise up to 80% by weight of the solids content of the dispersion. If the fat used is solid or semi-solid at room temperature, it is melted prior to use in forming the aqueous dispersion.

The hydrophilic film forming material which may be used in the aqueous dispersion includes any of the edible film forming materials well known in the art as encapsulating agents in the production of dried emulsion products. Suitable materials have the properties of good film forming capability on oily surfaces, low hygroscopicity, low viscosity, and preferably, emulsion stabilizing properties so that a separate emulsifying agent need not be included in the aqueous dispersion. Examples of suitable film forming substances include proteinaceous hydrophilic colloids such as sodium or calcium caseinate, whey solids, non-fat milk solids, gelatin, and the like; hydrocolloid gums such as gum arabic, gum tragacanth, guar gum, carboxymethylcellulose, methylcellulose, and the like; gelatinized starch, dextrins and chemically modified dextrinized starches. In general, it is preferred to use sodium caseinate, gum arabic and/or chemically modified dextrinized starches as the film forming material in the present invention, for such materials have all of the properties set forth above, including good emulsion stabilizing properties, so that a separate emulsifier need not be included in the aqueous dispersion.

The amount of film forming material included in the aqueous dispersion should be sufficient to provide a continuous film encapsulating the fat globules in the emulsion. This amount will depend on the specific film former used, the amount of fat in the aqueous emulsion, and the desired size of the fat globules in the dried emulsion product. Thus, any ratio of fat to film former is suitable which will result in the formation of a stable emulsion in which the fat globules are encapsulated with a continuous film of the film forming material. As the ratio is increased, a point is reached where a stable emulsion cannot be formed because of the lack of an adequate amount of film former, resulting in coalescence, agglomeration and rising to the surface (creaming) of the fat particles prior to drying of the emulsion concentrate. This point will vary according to the film former used. For example, with a sodium caseinate-butterfat system, a fat to sodium caseinate ratio of about 15 to 1 in the aqueous dispersion appears to be the upper limit in providing a stable emulsion. Although this limit cannot be set forth for every conceivable system, it can be readily determined in a given system by slowly increasing the ratio until a stable emulsion can no longer be formed. The lower limit of this ratio is controlled only by cost considerations and the desired size of the fat globules in the emulsion. That is, as the ratio is decreased, the size of the fat globules decreases. The fat globules in the emulsion preferably have an average particle size of less than about 10 microns. In general, it is preferred that the fat to film former ratio in the aqueous dispersion be in the range of about 2:1 to 20:1. When sodium caseinate is used as the film forming material in both stages, it is preferred that the ratio of fat to caseinate in the aqueous dispersion be in range of about 5:1 to 15:1, with an equivalent amount being added to the liquid emulsion concentrate after formation of the emulsion.

If the film forming agent used does not have good emulsifying properties, a conventional emulsifier may be added to the aqueous dispersion to increase the ease of formation of the emulsion and to promote stability of the liquid emulsion concentrate to be dried. Emulsifiers which may be used are those which are approved for use in foods, such as mono- and diglycerides, glycerol mono-stearates, sorbitan esters of hexitol anhydrides, polyoxyethylene sorbitan esters of hexitol anhydrides, and combinations thereof. It an emulsifier is used, it is typically present in an amount of about 0.2% to 2.0% by weight of the solids content of the emulsion concentrate.

A carbohydrate material, preferably a disaccharide, such as sucrose, lactose, maltose, and mixtures thereof, is included in the aqueous dispersion in an amount of from about 3% to 35% by weight of the solids content of the aqueous dispersion in order to promote emulsion stability of the dried fat emulsion product.

Other edible materials usually used in the production of dried fat emulsion products for nutritive or organoleptic purposes, such as, for example, coloring, flavoring, vitamins, minerals, preservatives antifoaming agents and the like, may if desired, also be included in the aqueous dispersion. If used, such

materials are usually present in small amounts.

In preparing the aqueous dispersion, the fat is melted (if a sold or semi-solid fat is used) by heating to about 55 °C. to 65 °C., and is added, with vigorous agitation, to hot water (65 °C.-90 °C.) in which the film forming material has been dispersed. The disaccharide is then added with agitation. If an emulsifier is used, it is usually added to the liquified fat. The amount of water is controlled to provide the aqueous dispersion with a solids content of about 25% to 75% by weight. The aqueous dispersion is then homogenized to an extent sufficient to provide an oil-in-water emulsion in which the fat globules have a desired particle size distribution, such as by homogenizing at about 140 to 210 Kg. per sq. cm. total pressure in a conventional two-stage homogenizer, with the fat globules in the emulsion being encapsulated in a layer of the film forming material.

After formation of the liquid emulsion concentrate, an additional quantity of hydrophilic film forming material is incorporated in the emulsion either prior to, during or subsequent to drying of the emulsion concentrate. The film forming material used in this second addition may be any of the film formers disclosed hereinabove, and may be the same as or different than the film former used in preparing the aqueous dispersion. The amount of film forming material in this second addition is substantially equivalent to the amount of film former contained in the aqueous dispersion. It is believed that this second addition of film forming material provides a second layer of film former encapsulating the fat globules which further protects the fat globules from oxidation, light, humidity and other deleterious conditions in storage. Such double encapsulated fat emulsion products exhibit better resistance to oxidation, particularly at elevated temperatures, than similar products prepared using an equivalent amount of film former in which all of the film forming material is added in a single step.

Thus, the second portion of the film forming material may be dispersed in a suitable quantity of water and added, with agitation, to the liquid emulsion concentrate, at any point prior to drying of the emulsion concentrate. Alternatively, the liquid emulsion concentrate may be dried, such as by spray drying, and the dried emulsion product instantized using water in which the second portion of the film former has been dispersed to wet the powder.

The dried emulsion product of this invention may be used in the production of any of the dry food systems in which dried emulsion products have been used in the past, such as, for example, dry cake mixes, powdered shortenings, dried coffee whiteners, topping mixes, sauce mixes, dried beverage mixes, and the like. Such products, when containing the dried emulsion product of the present invention, have improved resistance to oxidation and the development of rancidity, particularly at elevated temperatures.

The following specific examples are intended to illustrate more fully the present invention without acting as a limitation on its scope. As used herein, all percentages, parts, ratios and proportions are by weight and all temperatures are in °C. unless otherwise stated or otherwise obvious herefrom to one ordinarily skilled in the art.

Example 1

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A dried fat emulsion product having the following formulation was prepared.

Ingredient	Percent
Fat (butterfat)	75.0
Sodium Caseinate	15.0
Lactose, crystalline	10.0

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In preparing the dried emulsion from these ingredients, one-half of the sodium caseinate (i.e. 7.5g) was added to 92.5g of hot water (83° C.) and mixed well in a Waring blender. The fat was melted and added at a temperature of 60° C., with vigorous agitation, to the sodium caseinate dispersion, after which the lactose was added and the aqueous dispersion mixed until homogeneous. The resulting dispersion was then homogenized in a conventional two-stage homogenizer at 175 Kg./sq.cm. and 35 Kg./sq.cm. for the first and second stages respectively to form a liquid emulsion concentrate having a solids content of 50%. Since sodium caseinate has good emulsifying properties in addition to encapsulating and film forming capability, it was not necessary to include a separate emulsifying agent in the aqueous dispersion. The liquid emulsion concentrate was then pumped to a Bowen spray drier for drying. Just prior to introduction of the emulsion

concentrate to the spray dryer nozzles, a dispersion of 7.5g sodium caseinate in 92.5g hot water (83°C.) was added to the liquid emulsion concentrate in a 1:1 ratio, and the resulting emulsion was spray dried to produce a dried fat emulsion having a moisture content of less than about 3%. The dried emulsion was characterized as comprising a matrix of water soluble constituents having as the dispersed phase therein discrete small globules of fat, with the fat having been encapsulated in two stages.

Example 2

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A dried fat emulsion product was prepared in accordance with the two-stage encapsulation process of the present invention and the properties of the product compared to those of a dried fat emulsion prepared by conventional single-stage encapsulation procedures. That is, a dried fat emulsion was prepared using the ingredients, amounts and procedure set out in Example 1, with the single exception that corn oil, rather than butterfat, was used as the fat component. The product produced by this two-stage encapsulation process was identified as Product A. Another dried emulsion product (Product B) was prepared using the same ingredients (i.e. corn oil, sodium caseinate, lactose and water) in the same amounts and the same procedure, with the exception that all of the sodium caseinate (15g) was added to the aqueous dispersion in a single step prior to formation of the emulsion. The oxidative stability of the dried products as well as that of unencapsulated corn oil (Product C) was then determined by the following test procedure.

A 10g sample of the dried emulsion product was introduced into a 500 ml flat bottom jar capped with a rubber septa, with glass cotton being used to disperse the product throughout the jar and maximize its exposure to oxygen. The jars were stored at 45°C., 50°C. and 60°C., and the headspace oxygen was determined for each jar by gas chromatography at two week intervals to determine the half-life of headspace oxygen in each jar, that is, the time required for the headspace oxygen in each jar to be reduced to 10.5% (half the original concentration). The results of this measurement are set out below in Table 1.

Table 1

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Product Tested	Half-life (weeks)		
	45° C.	50 °C.	60°C.
Α	11	6.5	2.5
В	11	5.5	2.0
С	4.5	2.0	0.5

These test results show that the dried emulsion product produced in accordance with the two-stage encapsulation process of this invention has improved resistance to oxidation at elevated temperatures.

The particle size of Products A and B were determined using a Nicomp 270 Submicron Particle Sizer. Product B (the single-stage encapsulated product) had a bimodal particle diameter size distribution peaking at 600 and 140 nanometers, while Product A (the two-stage encapsulated product) had the same distribution with peaks at 1,125 and 240 nanometers.

Example 3

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A dried fat emulsion product having the following formulation was prepared

Percent
80.0
10.7
9.3

5

15

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The procedure set out in Example 1 was used in preparing this product. That is, one-half (5.35g) of the sodium caseinate was included in an aqueous dispersion containing the fat, lactose and sufficient water to provide a liquid emulsion concentrate having a solids content of 50% by weight. The remaining 5.35g of sodium caseinate was dispersed in water and added to the emulsion prior to spray drying. The dried fat emulsion had excellent resistance to oxidation at elevated storage temperatures.

Example 4

Five samples of dried fat emulsion products were prepared and evaluated using gas chromatography to determine the differences in the extent of oxidation between the products. All of the samples were prepared using a formulation containing 75.0g corn oil, 15.0g film forming material, and 10.0g lactose. Product samples D, E and F were prepared using the two-stage encapsulation procedure of Example 1. In preparing sample D, gum arabic was used as the film forming material, with 7.5g of gum arabic being included in the aqueous dispersion, and 7.5g being added to the emulsion prior to spray drying. Sample E was prepared using sodium caseinate as the film forming material, with the caseinate being added in two stages of 7.5 g in each stage in accordance with the procedure of Example 1. Sample F was prepared by including 7.5g sodium caseinate in the aqueous dispersion, and adding 7.5g gum arabic to the emulsion concentrate prior to spray drying. Product samples G and H were prepared by a single stage encapsulation method in which all of the film former was included in the aqueous dispersion. That is, in sample G, 15g of sodium caseinate was added to the dispersion, and in Sample H, 15g of gum arabic was included in the aqueous dispersion.

The spray dried emulsion concentrates of each sample were evaluated by gas chromatography to determine the extent of oxidation of the samples. That is, 5g of the sample being evaluated were purged using a Tekmar 4000 Headspace Concentrator. The samples were purged with helium (zero grade) at a flow rate of 80 ml/min. for 20 minutes at 75° C. The flavor volatiles were trapped on a Tenax porous polymer trap. After completion of the purge cycle, the carrier gas (ultrapure hydrogen) transferred the compounds onto the head of a DB-5 fused silica column held in a Hewlett-Packard 5890 gas chromatograph. During a 5 minute desorb cycle the column was held at -20° C. After that time, the oven temperature was ramped to 200° C. at the rate of 8° C. per minute. Components eluting from the column were detected by flame ionization and the total volatiles eluted from the column for each sample was determined by calculating the total peak area of the gas chromatographic profile for each example. The results of these tests are set out in Table 2.

Table 2

Product Sample	Film Forming Material(s)	Total Volatiles
D E F G	gum arabic/gum arabic sodium caseinate/sodium caseinate sodium caseinate/gum arabic sodium caseinate gum arabic	60,424,800 75,256,100 78,363,400 209,199,700 76,017,400

Since an increase in volatiles eluting from the column is indicative of increased fat oxidization, it is apparent that the two-stage encapsulation process of this invention provides increased resistance to oxidation. For example, Sample E (two-stage addition of sodium caseinate) had about 64% less volatiles than Sample G (one-stage addition of the same total amount of sodium caseinate), and Sample D (two-stage addition of

gum arabic) had about 20% less volatiles than Sample H (one-stage addition of the same amount of gum arabic).

Claims

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- 1. The method of producing a dried fat emulsion product having long term stability against oxidation which comprises
- emulsifying an aqueous dispersion containing an edible fat, a hydrophilic film forming material, a carbohydrate and water to form a stable liquid emulsion concentrate containing fat globules encapsulated with said film forming material,
- adding to said liquid emulsion concentrate a hydrophilic film forming material in an amount substantially equal to that contained in the aqueous dispersion, and drying said emulsion concentrate to provide a dried fat emulsion product.
- 2. The method defined in claim 1 in which the amount of film forming material included in the aqueous dispersion is sufficient to provide a continuous film encapsulating the fat globules.
- 3. The method defined in claim 1 in which the aqueous dispersion contains a ratio of fat to film forming material in the range of between about 2:1 to 20:1.
- 4. The method defined in claim 1 in which the fat comprises the major component of the solids content of the aqueous dispersion.
- 5. The method defined in claims 1 in which the film forming material is selected from the group consisting of proteinaceous hydrophilic colloids, hydrocolloid gums, gelatinized starch, dextrins and chemically modified dextrinized starches.
- 6. The method defined in claim 1 in which the film forming material added to the liquid emulsion concentrate is the same film forming material included in the aqueous dispersion.
- 7. The method defined in claim 1 in which the film forming material added to the liquid emulsion concentrate is different than the film forming material included in the aqueous dispersion.
- 8. The method defined in claim 5 in which the film forming material is selected from the group consisting of caseinates, gum arabic, chemically modified dextrinized starches, and combinations thereof.
- 9. The method defined in claim 8 in which the film forming material is sodium caseinate and is present in the aqueous dispersion in a ratio of fat to sodium caseinate of about 5:1 to 15:1.
- 10. The method defined in claim 1 in which the carbohydrate is a disaccharide which is present in an amount of from about 3% to 35% by weight of the solids content of the aqueous dispersion.
- 11. The method defined in claim 1 in which the fat globules in the liquid emulsion concentrate have an average particle size of less than about 10 microns.
- 12. The method defined in claim 1 in which the liquid emulsion concentrate has a solids content of about 25% to 75% by weight.
 - 13. The method deferred in claim 1 in which the aqueous dispersion contains an emulsifying agent.
- 14. The method of producing a dried fat emulsion product having long term stability against oxidation which comprises
- forming an aqueous dispersion containing an edible fat, a hydrophilic film forming material, a carbohydrate and sufficient water to provide an aqueous dispersion having a solids content of between about 25% to 75% by weight,
- emulsifying said aqueous dispersion to form a stable liquid emulsion concentrate containing fat globules encapsulated with said film forming material,
- drying said liquid emulsion concentrate to form a dried fat emulsion in which the fat globules have an average particle size of less than about 10 microns, and
- instantizing the dried fat emulsion particles with an aqueous medium containing an amount of the hydrophilic film forming material substantially equal to that contained in the aqueous dispersion.
 - 15. The product produced by the method of claim 1.

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EUROPEAN SEARCH REPORT

EP 90 10 1038

	DOCUMENTS CONSID			G ASSESSATION OF THE
ategory	Citation of document with ind of relevant pass	ication, where appropriate, ages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
χ	US-A-3 968 261 (L.P * Column 2, line 26 20; ex. *	. GOODMAN) - column 4, line	1-5,7- 13,15	A 23 D 9/00 A 23 C 15/14 A 21 D 2/16
A	PATENT ABSTRACTS OF 161 (C-352)[2217], 1 JP-A-61 15 733 (MIYO 23-01-1986 * Abstract *	Oth June 1986; & 📑	1,2,4,5	
A	US-A-3 989 852 (E. * Example 1; column	PALMER) 2, lines 10-43 *	6	
A	GB-A-1 113 462 (GEN * Claim 1; example 1	IERAL FOODS)	1-5,8- 13,15	
A	PATENT ABSTRACTS OF 108 (C-280)[1831], 1 JP-A-60 2173 (KUNOOF 08-01-1985	l1th May 1985; &	14	,
i	* Abstract *			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
				A 23 P
				A 23 C A 23 D A 21 D
-	The present search report has l	neen drawn im for all claims		
	Place of search	Date of completion of the se		Examiner
Т	HE HAGUE	12-09-1990	VU	IILLAMY V.M.L.
THE HAGUE CATEGORY OF CITED DOCUMENTS X: particularly relevant if taken alone Y: particularly relevant if combined with another document of the same category A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: earlier patent document, but published on, or after the filing date D: document cited in the application L: document cited for other reasons &: member of the same patent family, corresponding document		published on, or ation ons		
A: technological background O: non-written disclosure C: P: intermediate document A: technological background &: member of the same patent family, corresponding document				amily, corresponding



CL	AIMS INCURRING FEES
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The oresen	t European patent application comprised at the time of filling more than ten claims.
	All claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for all claims.
	Only part of the claims fees have been paid within the prescribed time limit. The present European search
	report has been drawn up for the first ten claims and for those claims for which claims fees have been paid.
	namely claims:
	No claims fees have been paid within the prescribed time limit. The present European search report has been
_	drawn up for the first ten claims.
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A	CK OF UNITY OF INVENTION
	Division considers that the present European patent application does not comply with the requirement of unity of id relates to several inventions or groups of inventions.
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□	All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
	Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search lees have been paid,
]	namely claims:
	None of the further search fees has been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims.



LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions, namely:

- 1. Claims 1-13,15: Process for producing a dried fat emulsion comprising the steps of emulsifying fat, water, carbohydrate and a film forming material, then adding to said emulsion a film forming material, and drying this emulsion (second addition of film forming material before or during drying).
- 2. Claim 14: Process comprising the steps of emulsifying fat, water, carbohydrate and a film forming material, drying this emulsion and instantizing the dried emulsion with an aqueous medium containing a film forming material (second addition of film forming material after drying).